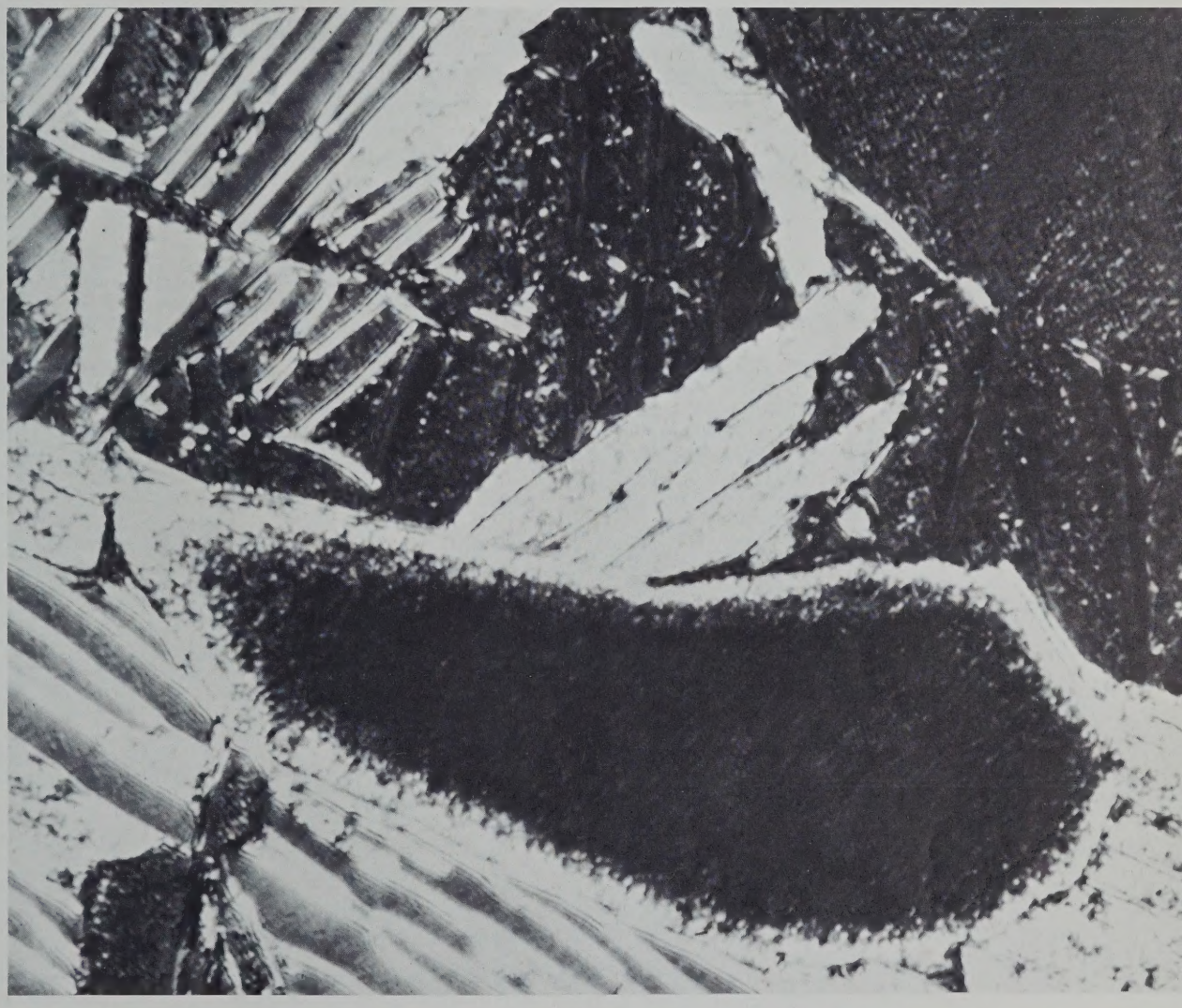
NATIONAL BUREAU OF STANDARDS · August / 1965

Technical News Bulletin



U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Technical News Bulletin

U.S. DEPARTMENT OF COMMERCE
John T. Connor, Secretary
NATIONAL BUREAU OF STANDARDS
A. V. Astin, Director

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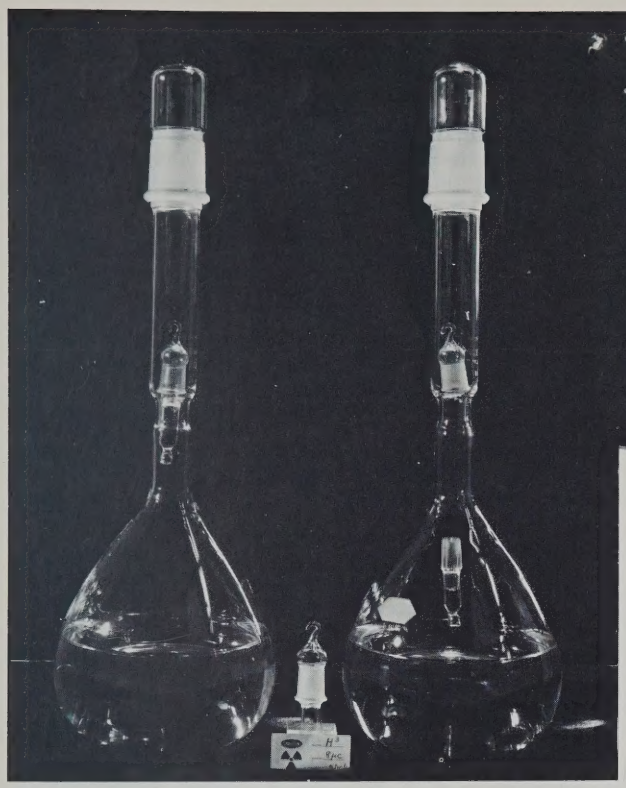
COVER: Micrograph of a titanium, 6-aluminum, 4-vanadium alloy specimen. The alloy was originally nitrided in an effort to impart the high surface hardness required for certain jet engine parts. This treatment, however, caused large grains to form. A second heat treatment then induced grain decomposition. The start of such decomposition, due to aluminum depletion, may be seen in the periphery of the large grain at bottom center. (*See story on page 123*)



Improved technique for calibrating

Radioactivity Standards

A study of the errors involved in the calibration of a tritiated-toluene radioactivity standard has led to several improvements in technique that may significantly increase the accuracy of such measurements. In particular, an extremely precise method of dilution was developed which is especially useful in diluting volatile liquids by a factor of 1000 or more. The study was carried out at the NBS Institute for Basic Standards, which has the responsibility for establishing radioactivity standards in this country.

S. B. Garfinkel, W. B. Mann, and R. W. Medlock of the Institute's radioactivity laboratory and O. Yura, a guest worker from the Electrotechnical Laboratory of Japan, recently prepared a tritiated-toluene standard in response to a demand for an additional standard to be used in the calibration of liquid scintillation counters.[1] In the process of calibrating the new standard (by intercomparison with an established tritiated-water radioactivity standard [2, 3]) it was found that vial variations and leakage, as well as weighing and dilution inaccuracies, were introducing errors which severely limited the accuracy of the measurement.

The calibration was carried out using the method of scintillation counting. A liquid scintillator (using toluene, containing alcohol, as the solvent) was prepared and divided into two equal portions. To one was added the tritiated-toluene sample and to the other was added the tritiated-water standard. Triple-distilled water, equal to the amount of tritiated-water standard used, was added to the toluene solution, and inactive toluene, equal to the amount of tritiated toluene used, was added to the water solution. The two solutions were thus chemically as nearly identical as possible and should therefore have been very similar in their quenching properties. Accurately weighed volumes of approximately 15 ml of the two solutions were pipetted into a number of 20-ml screw-cap vials.

Above: *Flasks used in new dilution technique. Left flask shows a sample bottle as it strikes constriction in neck of flask. In right flask, bottom half of sample bottle drops into the diluent.*

A test showed that differences as great as 3½ percent existed in the counting rate per unit mass of solution from vial to vial. It was also found that merely rotating the vial could change the counting rate by as much as 1 percent. To reduce the "rotation" error, it was decided to use a large number of vials and to count each sample for a large number of relatively short periods of time.

The effect of the "vial-to-vial" error was reduced by making a complete interchange of vials. Thus, when the first intercomparison had been completed all of the vials were cleaned and the experiment was repeated, this time placing tritiated-water samples in the vials which previously contained tritiated toluene and *vice versa*. A blank experiment was carried out with inactive liquid scintillator, showing that the vials could be washed free of radioactivity using toluene, ethanol, and ether, and thus that no radioactive "memory" persisted from one experiment to the other.

The liquid scintillation counting was done at about 0 °C to take advantage of the enhanced signal-to-noise ratio in this temperature region. It was found that the seals on many of the vials were leaking, and at the reduced temperature the pressure in the vials decreased while the oxygen solubility increased. These factors gave rise to increased oxygen quenching which resulted in a decrease in the counting rate of the sample.

The leaking was traced to non-uniform vial rims and to the tinfoil and cork vial-cap seals. To correct this, the vial rims were ground flat and teflon seals, lightly smeared with silicone grease, were used instead. Tests were conducted to determine whether the grease created a quenching effect, and the results indicated that if quenching took place at all, it was less than one percent. To eliminate the possibility of unequal quenching, the liquid scintillator was allowed to stand in contact with silicone grease for more than 12 hours, so that it would

saturate with respect to the grease. In actual fact, no observable amount of grease appeared to be dissolved.

Further tests showed that the method of weighing and also the evaporative losses of sample and solvent, in transferring the tritiated-toluene sample into the flask containing the liquid scintillator, were sources of substantial error. Even the displacement of toluene vapor on adding the sample to the flask could cause an error of the order of 0.1 percent, because of the high molecular weight of toluene. The activities of the tritiated water and tritiated toluene were such, that a dilution of the tritiated toluene in the liquid scintillator should be of the order of 1: 1000 for suitable count rates. To achieve this order of dilution with the required accuracy a better technique for mixing and weighing had to be devised.

Two large flasks, with constrictions in their necks, and two smaller sample bottles were therefore designed so that the bottle would pass through the neck of the flask but the bottle cap would not. Ball bearings were sealed in glass to the bottom of each sample bottle so that using a magnet, the bottle could be held in any position in the neck of the flask. The two large flasks are weighed and equal amounts of prepared liquid scintillator placed in each. The samples of tritiated toluene (approximately 1.5 ml) and tritiated water (approximately 0.5 ml) are enclosed in the sample bottles, and each bottle is weighed for 10 minutes to determine that there is no loss by evaporation. The appropriate flask containing the liquid scintillator is then opened, the sample bottle inserted and held at the top of the neck of the flask by means of a strong magnet, and the flask then closed. When the magnet is removed the sample bottle falls until its cap is stopped by the constricted neck of the flask. At this point the bottle and cap separate, the bottle falling into the liquid scintillator and the cap remaining wedged in the neck of the flask. The bottom of the flask is then cooled in an ice bath to condense any drops of liquid that might have splashed into the neck.

After about 3 hours the flasks are weighed to determine the mass of the liquid scintillator solution and the dilution factor, the mass of the sample having been previously determined by weighing the sample bottle both empty and while containing the sample.

The new dilution procedure improved the reproducibility of the different sets of measurements by at least an order of magnitude. Three independent measurements, starting with fresh scintillator solution and tritiated samples, gave a standard error of 0.09 percent.

A subsidiary experiment was carried out to determine whether there was any isotopic change due to evaporation of the tritiated-toluene standards. One such standard, that had been allowed to evaporate at room temperature to half of its original volume, showed no change in specific activity from that of a freshly opened standard.

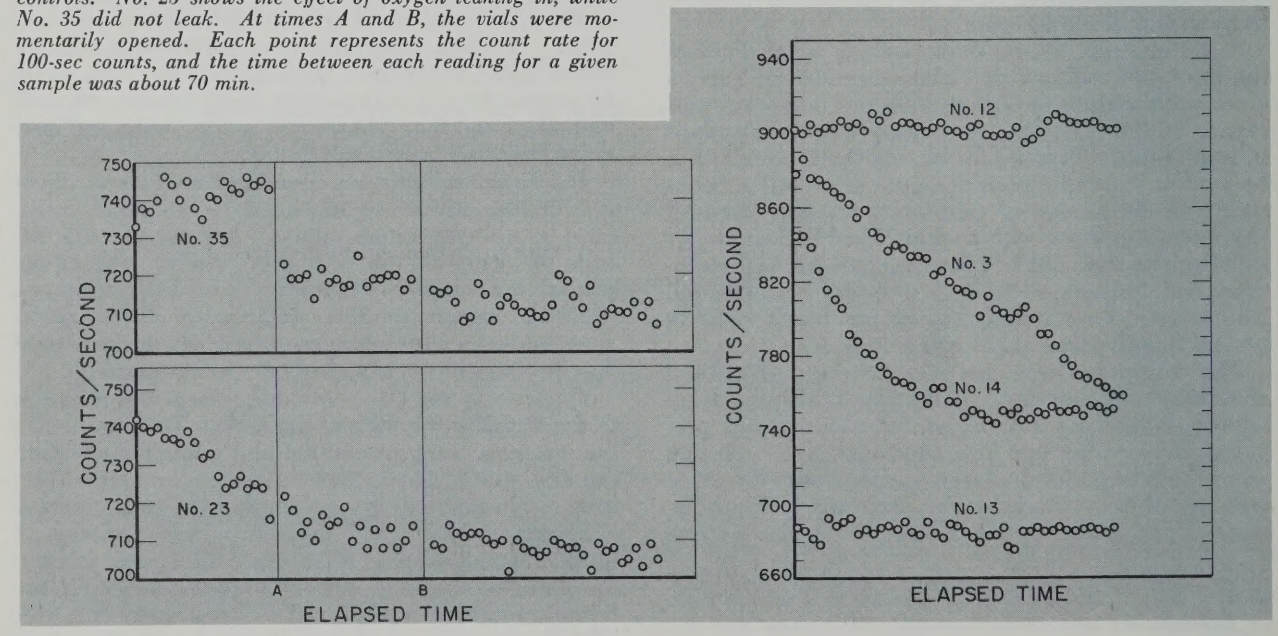
[1] *For further technical details see,* The calibration of the National Bureau of Standards' tritiated toluene standard of radioactivity, *by S. B. Garfinkel, W. B. Mann, R. W. Medlock, and O. Yura, International Journal of Applied Radiation and Isotopes,* **16,** *27 (1965).*

[2] A recalibration of the National Bureau of Standards tritiated water standards by gas counting, *by W. B. Mann, R. W. Medlock, and O. Yura, International Journal of Applied Radiation and Isotopes,* **15,** *351 (1964).*

[3] The National Bureau of Standards tritiated water standards, *by W. B. Mann and A. Spernol, International Journal of Applied Radiation and Isotopes,* **15,** *628 (1964).*

Typical count-rate data on 2 vials selected from 35 vials and controls. No. 23 shows the effect of oxygen leaking in, while No. 35 did not leak. At times A and B, the vials were momentarily opened. Each point represents the count rate for 100-sec counts, and the time between each reading for a given sample was about 70 min.

Typical count-rate data on 3 vials and on a flame-sealed ampoule (No. 13) used as control, selected from 15 vials and controls. The 3 vials had previously been flushed with nitrogen. No. 3 had a slow leak due to a faulty seal. No. 12 did not leak. No. 14 had a small hole drilled in the cap to provide a rapid leak. Each point represents the count rate for 100-sec counts, and the time between each point was about 35 min.



Microstructure of NITRIDED TITANIUM ALLOY

IN A RECENT NBS Institute for Materials Research study, the columnar grains induced in the titanium—6 aluminum—4 vanadium alloy by nitriding were investigated. The impact resistance of this widely used alloy is seriously impaired by the formation of such grains. Results of the work, conducted by Harvey Yakowitz and J. R. Cuthill of the NBS staff, show that the aluminum present in the alloy is responsible for columnar grain formation.[1] Experimental data obtained from the study may be useful in developing alloys with improved strength characteristics.

Both commercially pure titanium and the titanium alloy were experimentally nitrided in earlier Bureau experiments in an effort to impart the high surface hardness that is required for certain jet engine parts.[2] The effort was successful in the case of the unalloyed material, but nitridation of the alloy reduced its toughness. Thermodynamic calculations suggested that the aluminum present played a role in the columnar grain formation found in the alloy, but confirmation was not obtained. The present work was therefore undertaken to investigate the composition of the nitrided alloy. Heat treatments were employed in this work to determine whether or not the grains could be removed.

Small specimens were cut from 0.050-in. thick sheets and then nitrided in purified nitrogen at 1800 °F for 48 hours. Subsequent inspection of specimen cross sections under a microscope disclosed not only columnar but also square-appearing grains. With an electron probe microanalyzer, the two types of grains were found to have different percentages of both vanadium and titanium.[3] An anodizing treatment [4] that imparts characteristic colors [5] to the various microconstituents of a material was then applied. This treatment also disclosed that the two grain types were indeed different species, with the columnar grains appearing pink and the square grains green in color. Furthermore, because of these color differences, fields in the matrix could be associated with columnar or square grains by means of visual comparison.

To ascertain the tendency of the alloy to form each grain type as well as the distribution of each type within the metal, quantitative metallography by the use of electronic computer techniques [6] was employed. The results showed that columnar grains, occupying 42.2 percent by volume of the nitrided specimens, apparently formed more readily than the square grains which occupied only 5.42 percent by volume. The composition of the matrix and of each grain type was also obtained by quantitative metallographic computer analysis.

After the composition of the nitrided specimens had been determined, they were reheated *in vacuo* at 1750 °F and held at that temperature for varying periods of time. Subsequent study of photomicrographs made of specimen cross sections showed that a great deal of isothermal reaction had occurred in both the square and columnar grains. By means of the color anodizing technique, decomposition products could be traced back to an original grain. After a 19-hr exposure period, the complete absence of columnar grains was noted, although some of the square grains remained.

Inspection of the photomicrographs revealed that in the decomposition of the columnar grains, a reaction zone formed in the periphery of the grain and this zone later disengaged from the grain in the form of discrete lamellar needles. This process repeated itself until, upon continued holding at temperature, the interior of the grain developed a definite subgrain structure. Finally the columnar grain tended to disintegrate along these subgrain boundaries.

Diffusion coefficients found in the literature indicate that aluminum tends to diffuse 10 times more quickly than nitrogen in the alpha phase of titanium. Furthermore, the results of the present study had shown that the aluminum, after 19 hours at 1750 °F, had completely diffused into the matrix, and that the mean nitrogen concentration in the alloy structure was 8.2 atomic percent. It therefore appears that the disintegration process of the columnar grains is diffusion-controlled, and that aluminum loss and not nitrogen loss causes grain decomposition.

It is concluded from the results that the nitrided alloy does not have an equilibrium structure. However, it appears that the additional heat treatment causes a tendency toward equilibrium through diffusion processes.

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[1] *For further technical details, see* A mechanism for the decomposition of columnar grains in nitrided Ti-6Al-4V alloy, *by Harvey Yakowitz and John R. Cuthill, Transactions of AIME (in press).*

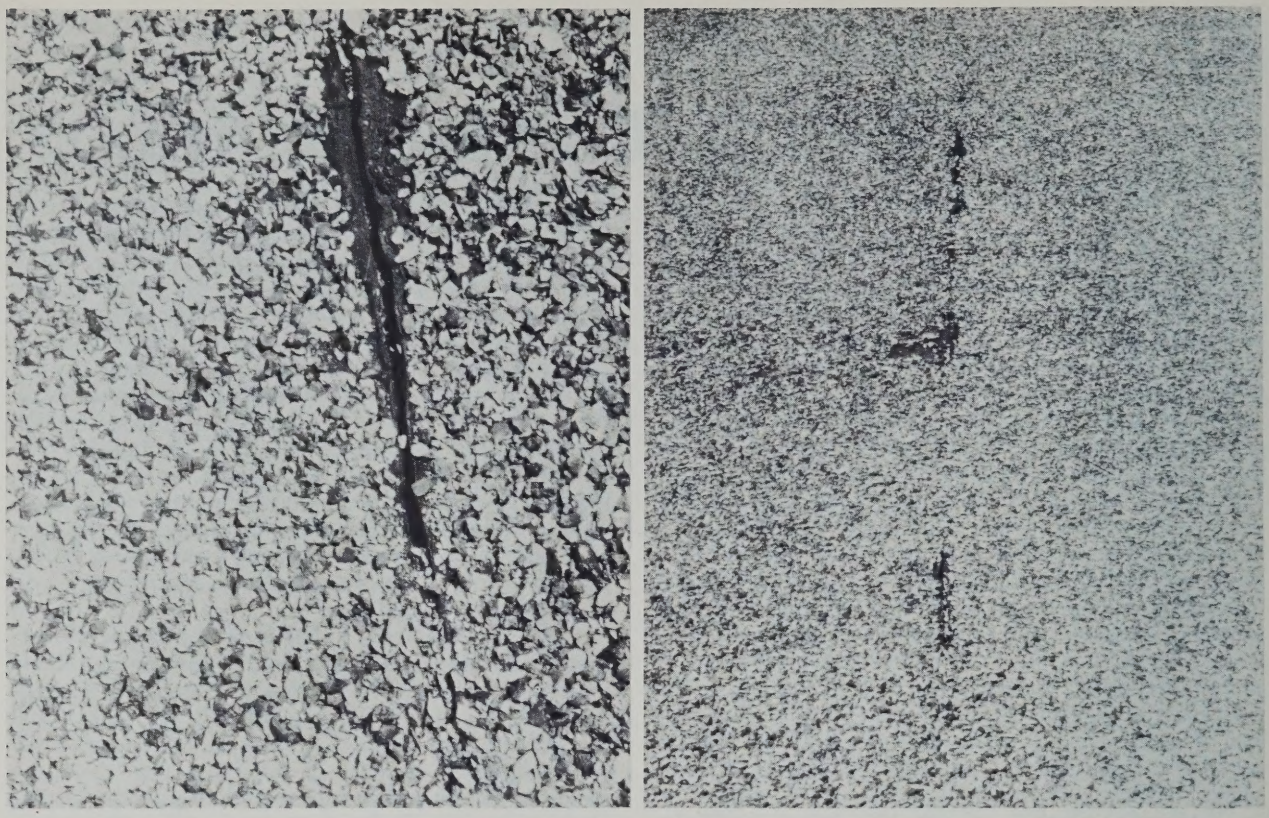
[2] Nitriding phenomena in titanium and the 6Al-4V titanium alloy, *by J. R. Cuthill, W. D. Hayes, and R. W. Seebold, J. Res. NBS* **64A** *(Phys. and Chem.) 119 (1960); also, NBS Tech. News Bull.* **44,** *90 (1960).*

[3] Metallurgical microanalysis with the electron probe, *by J. R. Cuthill, L. L. Wyman, H. Yakowitz, J. of Metals of the Metallurgical Society of AIME* **15,** *763 (Oct. 1963).*

[4] Anodizing as a metallographic technique for zirconium base alloys, *by M. L. Picklesimer, U.S. Atomic Energy Commission ORNL–2296 (1957).*

[5] Intermediate phases in superconducting niobium-tin alloys, *by L. L. Wyman, J. R. Cuthill, G. A. Moore, J. J. Park, and H. Yakowitz, J. Res. NBS* **66A** *(Phys. and Chem.) -351 (1962); also,* Constitution diagram for the niobium-tin system, *NBS Tech. News Bull.* **46,** *178 (1962).*

[6] Quantitative metallography with a digital computer: Application to a Nb-Sn superconducting wire, *by G. A. Moore and L. L. Wyman, J. Res. NBS* **67A** *(Phys. and Chem.) 127 (1963); also,* Digital computer used for quantitative metallographic analyses, *NBS Tech. News Bull.* **47,** *26 (1963).*



Reduce Splitting Failures of Built-Up Roofs

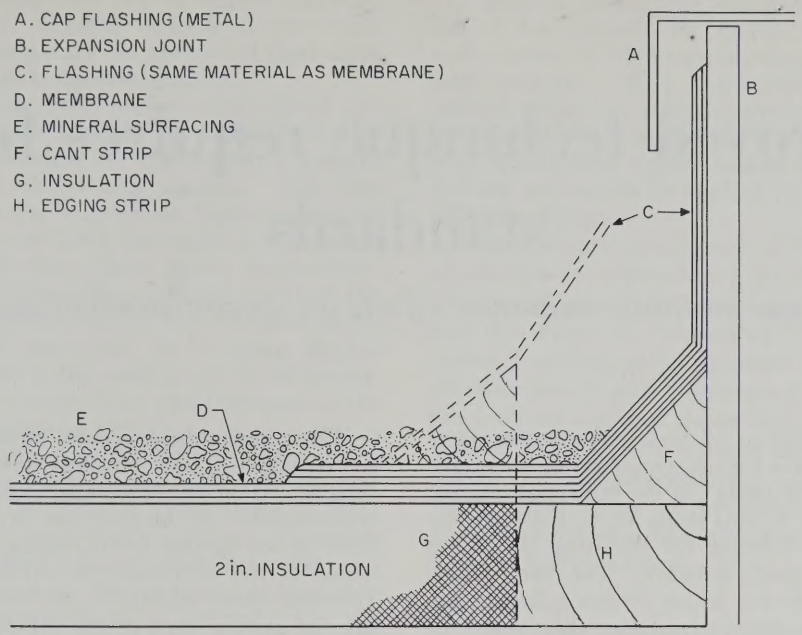
ASPHALT AND COAL-TAR products, the most widely used roofing materials in the United States today, accounted for approximately 1.7 billion sq ft of bituminous built-up roof construction in 1963 at a cost of about $450 million. Such roofing should have a life expectancy of 20 years or more; however, this life span is not always attained because of frequent premature failures of one or more components in the system.

The NBS Institute for Applied Technology conducts a continuing program to improve the effective use of building materials. In this program, a recent investigation sponsored by the Army, Navy, and Air Force was made to determine the cause of splitting failures of built-up roofs and to devise means to reduce the incidence of these failures. The results of the study [1] by William C. Cullen show that splitting in the waterproof layer (membrane) is caused by stress concentrations which are built up over insulation joints by the cumulative movement of both the insulation and the membrane. Also, the incidence of splitting failures can be reduced by making certain innovations in roof design and roofing procedures.

Above: *Typical splitting failure (left) and a line of failures over an insulation joint (right).*

In a previous study [2] of premature failures in built-up roof systems, splitting had been postulated to result from thermal shrinkage of the roof membrane. Therefore, in the present work the effects of thermal shrinkage, i.e., contraction due to decreasing temperature, were investigated. Tests showed that the coefficient of linear thermal expansion of the membrane was greater than that of the bituminous saturated, reinforcing felts from which it was constructed, but less than that of the bitumen used as the plying cement. In addition, the linear thermal expansion coefficient of both the saturated felts and the composite membrane were greater in the "across machine" direction than in the "in machine" direction of the felt, and that the tensile strength was generally greater in the "in machine" direction.

Relating thermal shrinkage to strength properties indicates that thermal shrinkage alone cannot produce the stress needed to split the membrane, although it is a factor that must be considered. Therefore, additional stress must combine with the shrinkage stress to produce a stress concentration of sufficient magnitude to cause a rupture. Field experience indicates that this additional stress may be produced when sections of insulation to which the membrane is secured draw apart. Using the same materials, steps may be



Dotted position indicates displacement of the flashing and cant strip caused by thermal shrinkage of the membrane in a built-up roof system.

taken to reduce the buildup of a stress concentration and thereby reduce splitting.

When insulation is used between the deck and the membrane, it should be placed so that the longitudinal (continuous) joint is parallel to the short dimension of the roof. The membrane should then be applied with the felts' "in machine" direction perpendicular to the continuous joint. Because the felts' "in machine" direction undergoes less thermal shrinkage than the "across machine" direction, less stress would thus be produced over the joint and because the felts' "in machine" direction is stronger, their ability to resist the stress without failure is increased. Taping of the insulation joints would tend to reduce the movement between units of the insulation and would further eliminate areas of stress concentrations over the joints. Also, the use of expansion joints (not structural) in the roofing membrane will reduce the magnitude of the stress produced by thermal shrinkage. The reduction of stress concentrations over the continuous joint between insulation boards should reduce splitting failures.

In securing the membrane to the insulation, the adhesive bond should be of optimum strength. It should be strong enough to withstand exposure conditions such as wind uplift, but sufficiently distributed on finite areas of contact to permit distribution of stress over large areas of the membrane in the event of thermal shock. This could be accomplished by spot, sprinkle, or strip mopping. The use of a laminated base sheet, in which both the elongation of the respective laminants and the strength of the adhesive bond between laminants could be controlled, may produce the desired effects.

Use of these suggestions should greatly reduce splitting failures and should increase the possibility of realizing the 20-year life expectancy of bituminous built-up roofs. The reduction of such failures would save the nation millions of dollars which are spent every year to repair and maintain built-up roof systems.

Although thermal shrinkage alone did not account for splitting failures, it is a frequent cause of the flashings pulling away from vertical surfaces. This type of roof failure can be reduced by fastening the flashings more securely to the parapet walls, expansion joints, and other vertical surfaces.

With the flashings securely fastened to the vertical surfaces, the stress set up in the membrane by thermal shrinkage of the membrane would increase. This increase would not, however, be of sufficient magnitude to cause splitting, particularly if the described innovations were incorporated into the roof design. Thus another type of failure common to built-up roof systems can be designed out of the system.

---

[1] *For further information, see* Effects of thermal shrinkage on built-up roofing, *by W. C. Cullen, NBS Monograph* **89** *(1965).*

[2] Premature failures of built-up roof systems, *NBS Tech. News Bull.* **47**, *No. 11, 203–205 (November 1963).*

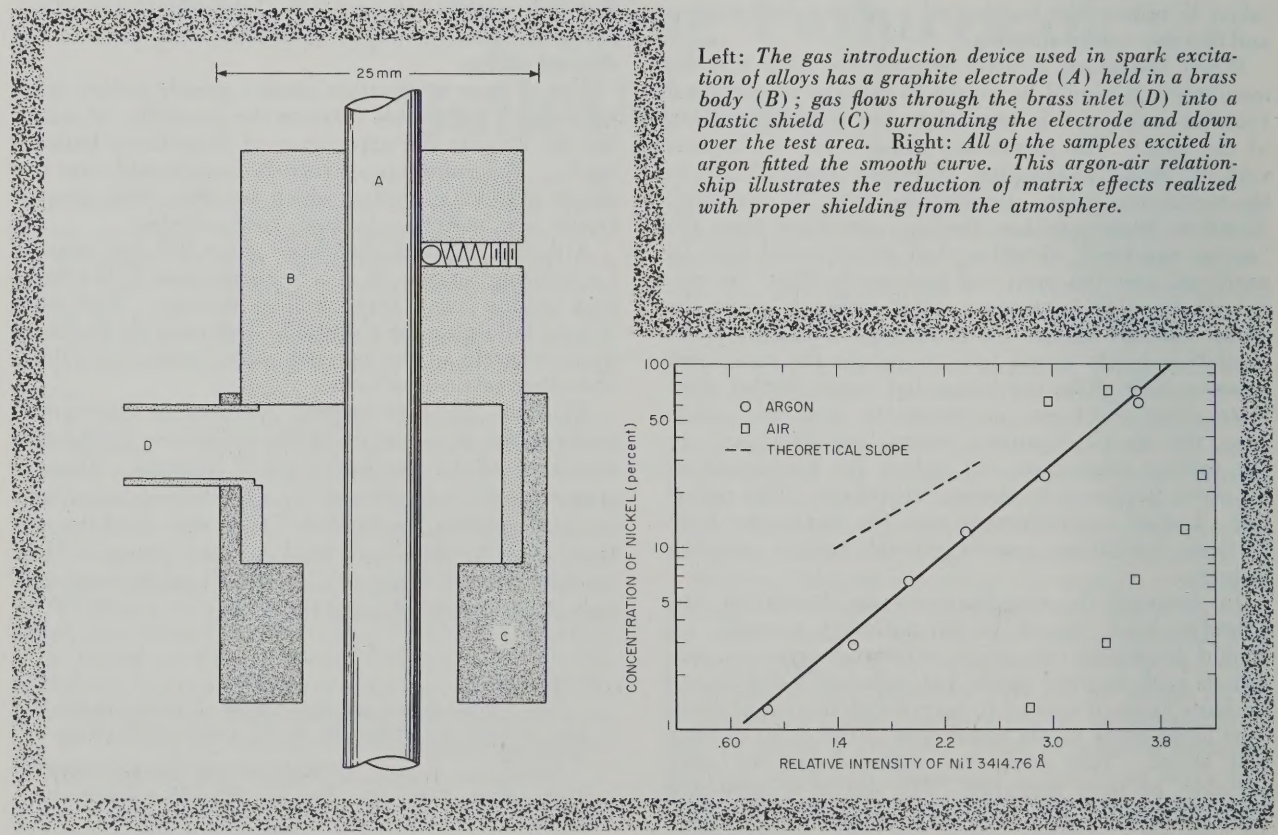
Improved technique requires fewer standards

Inert gas prohibits oxidation effects on spectrochemical analysis

Spark excitation of high-temperature alloys in selected atmospheres for spectrometric analysis was the subject of a recent study [1] at the NBS Institute for Materials Research. In this study H. C. Dilworth developed an improved device for shielding the alloys with an inert gas during excitation. The results show that the use of this device, which directs a flow of gas over the sample, can effectively reduce the number of primary standards needed to calibrate spectrometric equipment. The shielding technique should be of considerable interest to industry as it is rapid, convenient, and readily adaptable to commercially available equipment.

When high-voltage sparks strike the surface of an alloy sample, they volatilize minute portions of the sample and excite the metallic vapor to produce spectra characteristic of the elements in the sample. Variations in the spectra occur during excitation in air because of the variations in oxidation of the sample. However, these variations can be eliminated by shielding the excited area of the sample with an inert gas.

Previous investigators,[2,3] using spark excitation in argon and nitrogen were able to fit a variety of ferrous base alloys to a common set of analytical curves of concentration versus spectral line intensity. This reduced the number of standards required to calibrate



Left: *The gas introduction device used in spark excitation of alloys has a graphite electrode (A) held in a brass body (B); gas flows through the brass inlet (D) into a plastic shield (C) surrounding the electrode and down over the test area.* Right: *All of the samples excited in argon fitted the smooth curve. This argon-air relationship illustrates the reduction of matrix effects realized with proper shielding from the atmosphere.*

the analytical equipment and thus eliminated the need for standards closely matched to each of the alloys. However, these investigators did not extend their work into the field of high-temperature alloys.

In the present study, NBS undertook to extend this research to determine if a reduction in the number of primary standards required for spectrometric analysis of high-temperature alloys was possible. This was undertaken because the number of high-temperature alloys now in use (over 200) has made it prohibitive to produce standards that closely match these alloys.

A device was developed that excluded air from the analytical gap by a gentle tubular flow of either argon or nitrogen over the test area. By stopping the gas flow, comparison tests in the ambient air could be performed. Also, the change from one inert gas to the other could be accomplished rapidly.

In spectrometric analysis, the ratio of intensity of the spectral line of an internal standard to the spectral line of the element being investigated serves to minimize effects of variations in test conditions. In selecting an internal standard a linear relationship between concentration and spectral line intensity is desirable over the range of compositions of interest. For the high-temperature alloy samples, the plot of chromium concentration versus line intensity produced a linear relationship (with theoretical slope) over the composition range of interest. Also, in relation to other elements, the concentration of chromium remained fairly constant. Therefore, chromium was selected as the internal standard.

Analytical curves were established for the determination of six elements, Ni, Fe, Ti, Nb, Zr, and Mn in a wide variety of high-temperature alloys in wrought or cast condition. With spark excitation in argon, the plotted points fit smooth curves in all cases. This demonstrates that analytical curves can be established with a relatively small number of reference samples and the curves can be applied to the analysis of many different alloys.

To investigate the accuracy of the method, a number of alloys were selected and divided into two groups. One group of samples served as calibration standards and the others as "unknowns." As the samples included low-alloy steels, stainless steels, and high-temperature alloys, either chromium or iron was used as the internal standard depending on the matrix. The results with argon compared favorably with the reported chemical analyses, and demonstrated the satisfactory application of the method employing a minimum number of calibration standards to the analysis of iron- and nickel-base alloys.

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[1] *For further information, see* Spectro-chemical analysis of high-temperature alloys by spark excitation in argon and nitrogen, *by Harold C. Dilworth, Symposium on X-ray and Optical Emission Analysis of High Temperature Alloys, ASTM Special Technical Publication (in press).*

[2] *R. Bartel and A. Goldblatt, Spectrochem. Acta* **9**, *227 (1957).*

[3] *A. Arrak, Spex Speaker,* **8**, *No 3 (1963).*

New NRL-NBS Film

Preparation of White Cast-Iron Stardards

INDUSTRIAL, TECHNICAL, and student groups will be interested in a new motion picture, *Preparation of White Cast-Iron Standards*, now available from the Bureau. The 15-minute film, in 16mm color with sound, was produced by the Naval Research Laboratory in cooperation with NBS, and is loaned free of charge through the Bureau.

White cast-iron standards, issued by the NBS Institute for Materials Research, are used by industry and research laboratories throughout the world in controlling the chemical composition and quality of cast-iron. The experimental foundry at the Naval Research Laboratory, with its capability for very precise work, was used for the casting of two new groups of the standards.

Vivid furnace sequences are included in the film's story of the melting and casting procedure. These are followed by scenes made at NBS showing homogeneity testing of the samples by optical emission and x-ray spectroscopic methods of analysis.

This is a colorful and interesting depiction of the making of one of the many NBS standards which aid the nation's industries and contribute significantly to technological and economic growth.

For information on free loan of prints, write to the Office of Technical Information and Publications, National Bureau of Standards, Washington, D.C., 20234.

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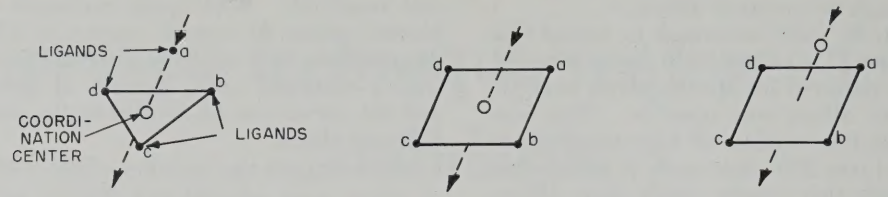
New chemical notation system



4A tetrahedron 4B tetragonal or square plane 4C tetragonal pyramid

As part of continuing NBS work in developing chemical information retrieval systems, Robert F. Pasternack of the NBS Institute for Applied Technology and Patricia M. McDonnell of the Patent Office have recently developed an alphanumeric notation system for inorganic coordination compounds which distinguishes among different geometric patterns of the same compound.[1] The system is based on a set of idealized reference structures and is intended to permit computer manipulation of three-dimensional structure information. Use of this system permits unambiguous designation of isomeric coordination compounds and may facilitate machine processing of information involving complex types of inorganic compounds.

The automation of patent searches is the general goal of a series of research projects sponsored by the Patent Office and carried out at the Institute's information technology laboratories and the Office of Research and Development of the Patent Office. NBS developments in this area include a rapid selector for automatic document retrieval,[2] remote stations for input to and output from a central computer, and computer routines for chemical structure searches in the HAYSTAQ [3] project.

The Patent Office's need for faster search techniques is increasing; applications are being received at a rate of about 300 per working day and have resulted in a backlog now numbering over 200,000 applications. Those dealing with chemical compounds constitute over 25 percent of the total.

A method of designating organic compounds for machine data processing was originated by H. W. Hayward [4] of the Patent Office and is now under development and revision by him in collaboration with S. J. Tauber of NBS. In a number of ways inorganic compounds present a greater problem because some are more complex in structure and the structures occur in a wider variety of geometric arrangements. The notation system devised for the inorganic coordination compounds by Dr. Pasternack and Miss McDonnell will make it possible to apply automatic search techniques to some inorganic structures also. Furthermore, the same principles are expected to prove useful in the extension of the notation system to other classes of inorganic compounds.

The new notation for coordination compounds is based on a table of reference structures which includes all geometric arrangements known to occur among inorganic coordination compounds. It can be shown by quantum mechanical considerations that only a limited number of structural types are possible. This permits the structure of any real coordination sphere to be classified as equivalent to one of a small number of idealized geometrical arrangements.

Twenty-two reference structures are used in describing all structures possible with one coordination center (central atom) and up to nine ligands (surrounding atoms or groups). Each reference structure is identified by a number (the coordination number or number of ligand attachments) and an uppercase letter identifying the geometric distribution of ligand attachments. The positions of the ligands about the coordination center are specified by lowercase letters called locant designators.

The appropriate reference structure identifiers and chemical symbols describing a compound are used to form an alphanumeric expression describing the structure. The ion $CoCl_4^{-2}$, for example, is translated into $[co\ni l,4A\in<<cl]1a,1b,1c,1d[>>4]2\theta$. Such terms may be transcribed for machine input via punched cards or punched tape: the machine can be programmed to seek and retrieve information on complete or partial structures.

Above: *The structural possibilities for an inorganic compound consisting of one coordination center and four ligands show three possible isomers. The alphanumeric designation system (4 for number of ligands, A, B, and C for different stereochemical configurations, and a, b, c, and d for the locant designators) facilitates encoding inorganic compounds for automatic patent searches.*

[1] A line-formula notation system for coordination compounds, *by P. M. McDonnell and R. F. Pasternack, J. Chem. Doc.* **5,** *56 (Jan. 1965) and* Designation of ligand positions in coordination complexes, *by R. F. Pasternack and P. M. McDonnell, Inorg. Chem.* **4,** *600 (April 1965). This work is related to a broader Federal effort, led by the NSF with participation by NIH and DOD. A major part of this work is carried out by Chemical Abstracts Service.*

[2] The rapid selector, an automatic document retrieval device, *NBS Tech. News Bull.* **43,** *178–179 (Oct. 1959).*

[3] Haystaq, a program for automatic searching of chemical literature, *NBS Tech. News Bull.* **43,** *192–193 (Oct. 1959).*

[4] A new sequential enumeration and line formula notation system for organic compounds, *by H. W. Hayward, Patent Office Research and Development Report No. 21 (Nov. 1961), available at cost of $1.25 from the Superintendent of Documents, Government Printing Office, Washington, D.C., 20402.*

Pressure Surge in Cryogenic Lines

Scientists at the NBS Institute for Materials Research have attacked the problem of predicting pressure surges in cryogenic transfer lines during cooldown as part of a study sponsored by NASA and the Air Force. Using an experimental apparatus equipped with a supply Dewar, flowmeter, pitot tubes, and pressure sensors, they were able to learn enough about the cooldown process to construct a mathematical model which simulates the initial portion of the cooldown process. W. Gene Steward of the Institute's Cryogenics Laboratory (Boulder, Colo.) described the computation of results from the model and comparison of results with experimental data in a paper presented at the 10th Annual Cryogenic Engineering Conference held Aug. 17–21, 1964, at the University of Pennsylvania, Philadelphia, Pa.[1]

Uses for cryogenic liquids flowing through transfer lines range from brain surgery to fueling rockets. Whether these transfer lines take the form of surgical cryoprobes or pipelines the size of city water-mains, they may have one common undesirable operating characteristic: a period of surging during cooldown. Cryogenic fluid, upon contacting the warm pipe, boils or flashes to a vapor, and the vapor evolution rate is greater than the vapor discharge rate. The result is a back pressure which slows or reverses the liquid flow. Even though the liquid flow may reverse, a certain amount of liquid is entrained in the vapor traveling downstream. This liquid contacts the warm pipe, produces still more vapor, and in turn causes even higher back pressures. The surging and high pressure may produce severe strains on lines, couplings, vents, and flow instruments.

The pressure recedes after part of the initial charge of liquid has boiled away or has been forced back into the supply vessel. After the initial surge, the process cycles with gradually diminishing pressure amplitudes until the pipe is completely cooled.

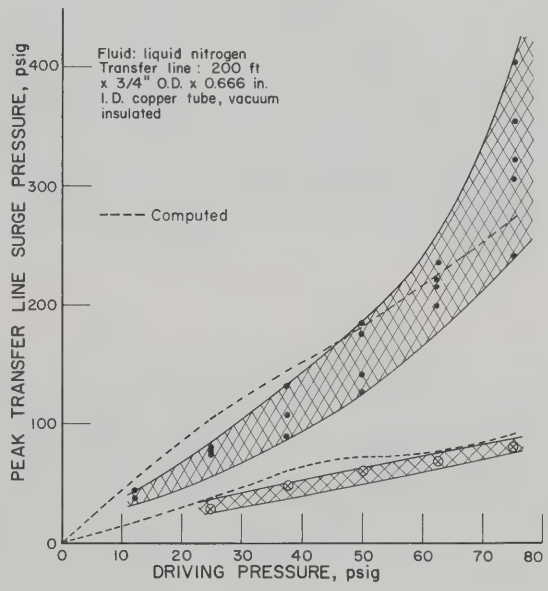
Actual flow processes in cooldown are extremely difficult to analyze as the process is unsteady; the two phases are very far from thermal equilibrium; the distribution of the liquid in the vapor is nonhomogeneous; and the mass, momentum, and heat transfer between the phases depend upon the unknown size and shape of the droplets as well as the relative velocities.

A grossly simplified hypothetical model of a transfer system used by NBS scientists enables them to compute theoretical data on peak surge pressures which are useful in the design of transfer systems. One principal assumption for the simplified model is that fluid interfaces are in one plane perpendicular to the cross section of the transfer line rather than considering a liquid-vapor mixture in the line.

Other characteristics of the model include the assumptions that the transfer line is constant in cross-sectional area, horizontal, and straight; that there are no flow restrictions other than the inlet valve and the friction of the pipe; and that there is no addition of heat to the pipe during the cooldown period. Fluid momentum, heat transfer, velocity change at liquid-vapor interface, and Fanno flow in the vapor stream are expressed as mathematical equations with variables in fluid properties, geometry, and in the flow and heat transfer expressions.

With this model NBS scientists, using an electronic computer, can calculate peak surge data for conditions described by any change in the variables. The graph shows a comparison between experimental and computed pressure. This comparison demonstrates that computed pressure history and flow rate are approximately correct. The data match similarly for varied initial conditions. Further work with other fluids and geometries will be required to determine whether the solution is very general in nature or is subject to limitations of application.

[1] Transfer line surge, *by W. Gene Steward, Advancements in Cryogenic Engineering* **10**, *Plenum Press*, New York, N.Y. (1965).



Experimental data and computed theoretical data for peak transfer line surge pressure versus inlet driving pressure were compared. Upper data points are for liquid entering at the normal boiling temperature. Lower points are for liquid entering in thermal equilibrium at the driving pressure. Valve opening time, approximately ½ second.

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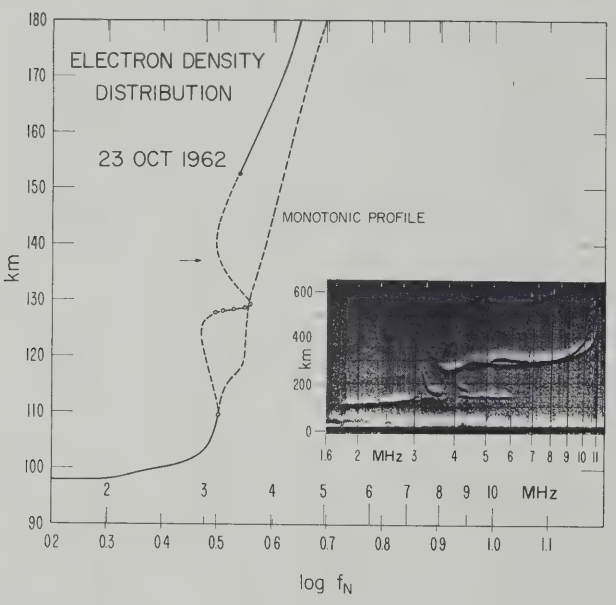


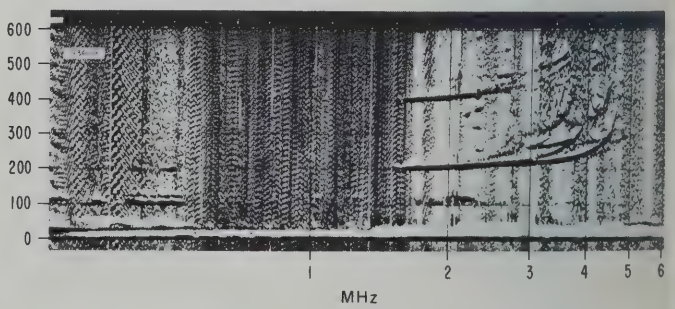
Top: *The ionogram being inspected by Irene Brophy shows the increasing heights from which radio signals of increasing frequency were reflected back to earth. The ionogram was recorded on 35-mm film in an NBS ionosonde; here it is projected for transcribing the data in numerical form.* Bottom: *Electron distribution just before Firefly Ethel, 1311 CST 23 October 1962. (A natural "ledge" of ionization exists at 129 km; 20 kg of SF_6 were released in the valley at 137 km.* Insert: *Ionogram from which N(h) profile was calculated.)*

The NBS Central Radio Propagation Laboratory (CRPL) has recently completed an analysis of ionospheric data from rocket flights to determine the effect of chemicals and explosions on the electron density of the ionosphere.[1] The data consist of ionosonde observations made during releases of chemicals and high explosives in the upper atmosphere in cooperation with the Air Force Cambridge Research Laboratories (AFCRL). The releases were made from a series of rockets launched at the Air Force Elgin (Fla.) Rocket Range during October, November, and December 1962. Ionosonde observations made during 12 chemical releases from 5 rockets were used by J. W. Wright of CRPL in reaching his findings.

Virtually all of the radio techniques over past years for ionospheric explorations are sensitive only to a "trace-element" in the upper atmosphere—that is, to the electrons that normally result from absorption of sunlight. Despite the significance attached to electrons because of their importance for radio communication, their concentration is, to a great extent, the incidental by-product of a complex of photo-chemical and chemical reactions. The complicated variability of the electron concentration, as observed by radio means, is largely a result of delicate shifts in the equilibria of a variety of reactions involving neutral and ionic species. Thus, relatively small changes in the temperature of the atmospheric constituents, or in the atomic and molecular concentrations, or the introduction of small amounts of new molecular species, can have quite disproportionate effects on the electron concentration.

In Project Firefly the CRPL scientists studied the constitution of the ionosphere by analyzing the alteration in the ionograms with localized changes caused by the release of chemicals at selected altitudes. An important use of these experiments is to determine the

Ionograms taken from Firefly Karen (15 Novembe



EASES IN THE PHERE

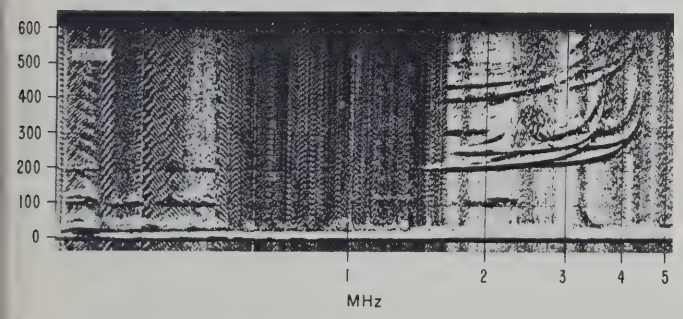
III Project

ambient electron distribution, $N(h)$, in the region into which the chemicals are released. The model C-4 ionosonde was modified to encompass an observing range from 0.25 to 20 MHz, and a high-gain log-periodic and switched dipole antenna system was designed to provide useful observations throughout this range. Without these modifications, reliable $N(h)$ profiles could not have been calculated.

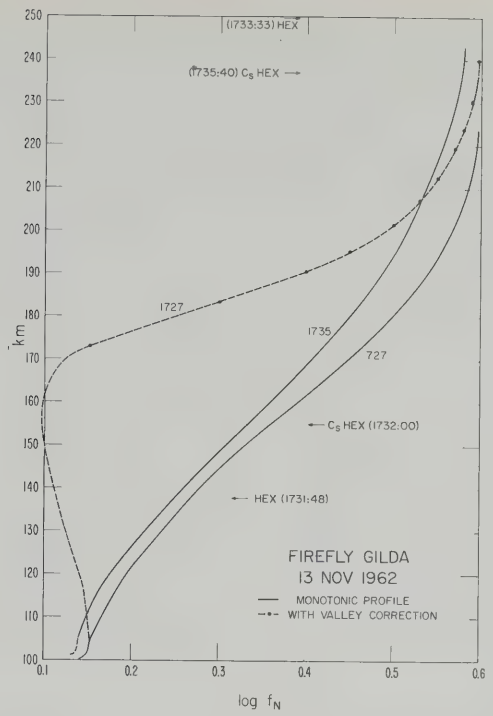
The releases fall into two categories: (1) those in which substances with high electron affinity (sulfur hexafluoride, SF_6) or those which might accelerate the natural electron loss processes (carbon dioxide, CO_2) were released in the *E* and *F* regions, and (2) those where detonation of a pure explosive (HEX) or cesium-salted high explosive (CsHEX) occurred in the *F* region. Three of the rockets made releases at preselected altitudes and two made only a single release as they rose in flight. The data were recorded as ionograms beginning before the launch and including all effects of the releases. The five launches were given feminine names: Ethel, Fanny, Gilda, Karen, and Martha.

Firefly Ethel

Firefly Ethel was designed to examine the properties of a region of reduced electron density suddenly produced in the normal daytime *E* layer by release of SF_6. The release occurred at the precise second the ionosonde passed through 4 MHz. No instantaneous effect was noticed, but the ionograms revealed a strong echo to 6 MHz at a slant range of 156 km. If this strong echo is considered to come from a region of increased electron density, the presence of such a region might be accounted for by "snowplow" action (a compression of ambient ionization by the expansion of the material), but other releases of inert material at the



F2) *following the release of SF_6 near the F2 peak.*



Top: *Electron distributions during Firefly Gilda, 1727 and 1735 CST, 13 November 1962. (The dashed curve represents a more probable profile. Heights of the various detonations are shown.)* Bottom: *Richard Lyons removes a magazine of exposed film from an NBS ionosonde. The 35-mm camera has recorded ionograms, presented on an internal oscilloscope, of frequency versus height from which the ionosonde signal was reflected.*

same altitude have not shown this effect. A more likely interpretation is that the SF_6 has removed electrons from within a bounded region, thereby causing a sharp discontinuity of the refractive index across the boundary. A similar experiment with the "Firefly 1960" chemical release series gave similar results. In that experiment the most definite effect of the release was again the production of an echo to frequencies exceeding the ambient plasma frequency. These experiments would appear to give proof that radio reflections resembling "Sporadic-*E*" echoes on ionograms can return from ionospheric regions of sharply reduced electron density.

Firefly Fanny

The four explosive releases of Firefly Fanny were intended to study the effects of shock waves in the *F* region. The addition of cesium to two of the charges was expected to enhance the effects of ionization *created* by the detonation. There has been some uncertainty in the past concerning the relative importance of ionization created in the shock front versus effects from the ambient ionization on radio Doppler observations during the first few milliseconds following the burst.

In this experiment, no effects were seen synchronous with burst 1 or 2, but prior to burst 3 an echo is seen beyond the *F* echo between 2 and 2.5 MHz. The real heights of reflection of these frequencies fall between 180 and 220 km. Thus, if burst 2 is responsible for this, it has sent ahead a disturbance which travels from 157 to approximately 200 km at the upward rocket velocity of 1 km/s. None of the new echoes can be seen to a great virtual height. This suggests that the disturbance so far is confined to altitudes below the *F* peak, expected from the positions of bursts 1 to 3. Following burst 4 an echo trace is seen which parallels throughout the ordinary and extraordinary echoes from the ambient *F* region.

While it is possible to attribute the final disturbances to one or another of the individual detonations, it is fairly clear that the aggregate effect was similar to the natural traveling ionospheric disturbances frequently seen on ionosonde recordings. Most of the energy of these detonations is ultimately dissipated in the form of heat; the heated region expands and the ion density within it becomes smaller, thereby producing a region of the kind required for these observations.

Firefly Gilda

The experiment of Firefly Gilda was similar to that of Fanny, except that it was conducted under sunlit conditions at the altitudes of detonation. No effect whatever can be seen from the first detonation. There is no evidence that the first burst of Fanny produced an *F*-region effect, either, so that the effects of a simple high explosive in regions of low electron density do not seem significant to these observations whether sunlit or not. The second burst, salted with cesium, produced a direct echo to the ionosonde.

No effects are synchronous with the 3d or 4th bursts, but a new type of *F*-region disturbance begins about one minute after burst 3, and develops strongly after burst 4. These bursts occur near or above the *F* peak. These new echoes are first seen at high frequencies and then later at lower frequencies, lower electron densities, and lower heights. The disturbance thus propagates downward through the *F* region.

As with Firefly Fanny, the echoes from the disturbance are those that could be expected from a region of reduced electron density, surrounded by an unaffected region. The presence of sunlight does not seem to have affected appreciably the lifetime of the disturbance. In both experiments, it is remarkable that no evidence of movement is seen; the slant range of the disturbance echoes remains essentially constant through the lifetime of the disturbance. This is in marked contrast with the well-known traveling behavior of the natural disturbances to which the echoes associated with these irregularities otherwise bear a strong resemblance.

Firefly Karen

Releases of CO_2 and SF_6 from Firefly Karen were intended to accomplish a reduction in *F*-region electron densities without the introduction of thermal energy. Carbon dioxide is released in great quantities in the exhaust of large rockets, and might accelerate the dissociative recombination process by which *F*-region electrons normally disappear through the reaction:

$$CO_2 + O^+ \rightarrow O_2^+ + CO$$

together with

$$O_2^+ + e \rightarrow O' + O''$$

The high electron affinity of SF_6 would be expected to lead to an even more marked reduction in electron density. The ionograms made during release 2 show a large disturbance caused by the SF_6 release.

It is puzzling to consider the process by which this region of reduced electron density returns to normal. As with the high-explosive releases, there is no evidence of rapid lateral translation of the region. The echoes of the region simply become weaker and disappear over a period of several minutes. A possible interpretation is that the region returns to normal in isolated pockets which gradually grow, leaving a diminishing outline of the disturbed region which finally cannot be detected.

Firefly Martha

One difficulty with the Firefly Karen electron-removal experiments is that the effects exist well beyond the observable lower limits of the nighttime *F* region; it is clear from the observations that large perturbations occur near and below the base of the *F* region where observations become more difficult because of strong broadcast-band interference to radio frequencies reflected there. A daytime experiment (Martha) was

therefore conducted, in which, despite a single large release of SF_6, the effects were expected to be confined at lower heights by rapid electron production and photo-detachment of SF_6 at approximately *F*1 layer levels.

The SF_6 was released just below the *F*2 peak, at 222 km. An effect was seen 7 seconds after the release. The sequence of events seems to be this: The SF_6, upon release, expands within at most a few seconds to a sphere of about 3 km diameter at ambient pressure, containing about 100 moles of gas. The cloud of SF_6 continues to expand laterally by diffusion, and falls vertically by diffusion and gravity. As long as the concentration of SF_6 considerably exceeds the ambient electron concentration, the latter will be appreciably reduced in the cloud because of the rapidity of the attachment process. Electrons from above the cloud diffuse rapidly into it, although only along the nearly vertical geomagnetic field, and at about 75 percent of the vertical rate ignoring the geomagnetic field. Thus, the hole becomes elongated along the magnetic field by electron-ion diffusion from above the cloud and develops to lower altitudes by vertical diffusion under gravity of the SF_6.

For the first several seconds, it appears that the SF_6 falls as a "snow" of conglomerated particles of mass larger than of a single molecule; this might result from its lowered temperature during the rapid initial expansion of the SF_6 cloud.

Name Date Launch, CST	ETHEL 23 Oct. 1310	FANNY 10 Nov. 1903	GILDA 13 Nov. 1730	KAREN 15 Nov. 1926	MARTHA 15 Dec. 1130
Release 1	SF_6	HEX	HEX	CO_2	SF_6
Kg.	20	5	5	5	22.7
Alt. Km.	137	140	137.7	234	222
Objective	N_e Removal	N_e Perturb.	N_e Perturb.	N_e Removal	N_e Removal
Release 2		CsHEX	CsHEX	SF_6	
Kg.		5	5	20	
Alt. Km.		157	155	225	
Objective		N_e Perturb.	N_e Perturb.	N_e Removal	
Release 3		HEX	HEX		
Kg.		5	5		
Alt. Km.		230	250		
Objective		N_e Perturb.	N_e Perturb.		
Release 4		CsHEX	CsHEX		
Kg.		5	5		
Alt. Km.		260	237		
Objective		N_e Perturb.	N_e Perturb.		

The efficiency and careful planning of the program of 27 rocket launchings by the Air Force Cambridge Research Laboratory, under the leadership of Dr. N. W. Rosenberg, was essential to the success of the observations by the participating agencies.

[1] Ionosonde studies of some chemical releases in the ionosphere, *by J. W. Wright, Radio Sci. J. Res. NBS* **68D**, *189–204 (Feb. 1964)*.

Radio Spectrum Utilization

Radio Spectrum Utilization, a revision and extension of the 1952 *Radio Spectrum Conservation*, has recently been published by the Institute of Electrical and Electronics Engineers.[1] This report of the Joint Technical Advisory Committee of the IEEE and the Electronic Industries Association is a major contribution to the scientific literature on spectrum conservation.

Chapter III, which constitutes the greater part of the book, was prepared by a subcommittee composed primarily of members of the staff of the Bureau's Central Radio Propagation Laboratory. It summarizes present knowledge of radio wave propagation in various portions of the radio spectrum and the effect of technical characteristics of transmitters, receivers, and antennas. Members of the subcommittee from CRPL are Jack W. Herbstreit (chairman), A. F. Barghausen, A. P. Barsis, H. V. Cottony, W. Q. Crichlow, D. D. Crombie, R. T. Disney, E. F. Florman, T. N. Gautier, G. W. Haydon, D. W. Patterson, P. L. Rice, Catherine Stephens, and C. C. Watterson.

The radio frequency spectrum is a major natural resource, which fortunately does not diminish with use as do material resources. Increasing use of the spectrum, however, has made its efficient use necessary in order to accommodate the needs of as many as possible of its users. *Radio Spectrum Utilization* is intended to help administrators achieve this goal by practicing spectrum conservation.

[1] *Available at a cost of $10 from the Institute of Electrical and Electronics Engineers, 345 East 47th Street, New York, N.Y., 10017.*

Effect of MOISTURE ON FIRE ENDURANCE OF PLASTER

Exposed surfaces of four pairs of specimens show the effect of typical fire tests. The right member of each pair was moist, the left was air dry. The thicknesses of the specimens were 3-in. (b), 1½-in. (c), ¾-in. (a), and ⅜-in. (d).

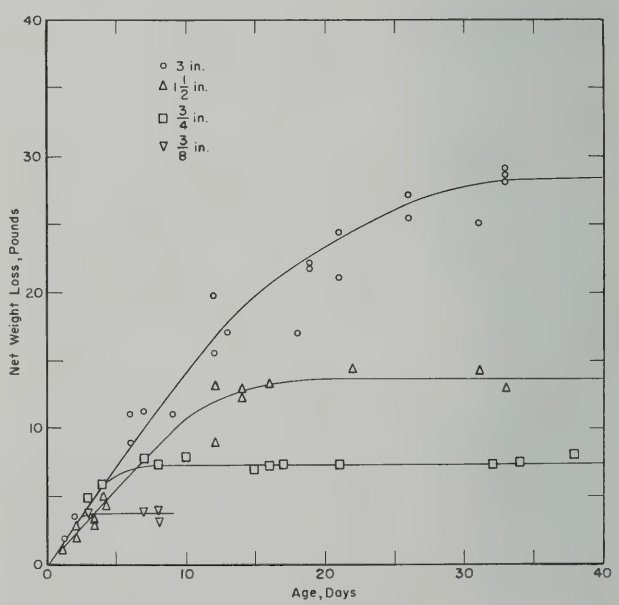
A study of the effect of moisture on fire endurance of lightweight-aggregate gypsum slabs has recently been completed at the NBS Institute for Applied Technology. The results of the study,[1] by J. V. Ryan, show that small amounts of free water in the plaster, as well as chemically combined water, have a significant effect on fire endurance.

It has been recognized for years that the moisture content of building material affects the fire endurance of building components and systems. The endurance of building components protected with gypsum plaster is due in a large part to the heat absorption of the chemically combined water in the plaster as it is released and evaporated. The purpose of this study was to determine the magnitude of the effect of free-moisture content on fire endurance.

Lightweight-aggregate gypsum plaster was chosen because it is about one-third to one-half water when the plasterer places it, but it eventually reaches a moisture content below one percent. A large part of the initial mix water goes into chemical combination, but even more remains as free water that evaporates until the moisture content reaches an equilibrium value. This value depends upon the temperature and relative humidity of the surrounding air, and the plaster-aggregate combination.

Plaster specimens were prepared in various thicknesses and then aged in a controlled atmosphere. During aging, the moisture contents and weight losses were determined at appropriate intervals.

The initial weight loss was at a constant rate, essentially the same for all thicknesses, followed by a period of decreasing rate until it leveled off at a very low rate near zero. Drying of the thickest specimen continued at the low rate for several months.



Weight losses of various gypsum plaster slabs plotted against age.

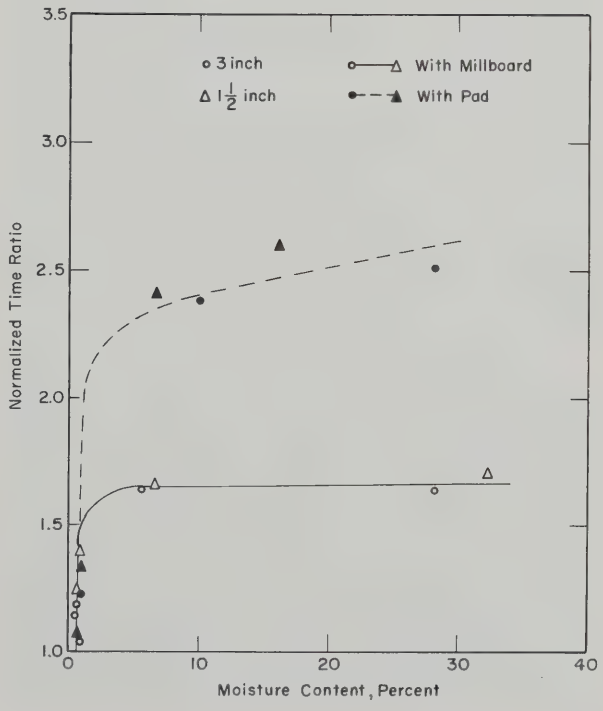
James V. Ryan monitors the progress of a fire test on a plaster slab. The slab is hidden by fire bricks and an asbestos millboard covering. Continuous water flow through the pan above the furnace provides a stable thermal background.

The moisture content was plotted against an age-thickness factor obtained by dividing the age of a specimen by its thickness. This age-thickness factor was used to express age data in terms of specimens one-inch thick. The data from specimens of all thicknesses fell close to a single curve indicating that the moisture content is related directly to age and inversely to thickness.

Specimens were fire tested on a small furnace regulated to the same time-temperature curve defined for large scale specimens. Fire endurance was judged as the time required to raise the specimen's unexposed-surface temperature 250 °F (139 °C) above the initial temperature. A plot of these times gave a curve for each thickness. To eliminate thickness as a variable, the times were normalized; that is, equivalent times computed for specimens one-inch thick. A plot of normalized times against the age-thickness factor gave a single curve illustrating the relationship between fire endurance and age.

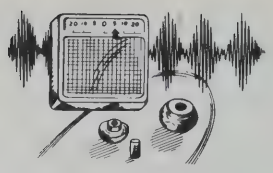
The effects of weight loss and moisture content on fire endurance were determined by plotting these variables against the ratio of normalized times to minimum normalized time (fire endurance of a 1-in. specimen in equilibrium with ambient air). The results showed very little decrease in fire endurance during the initial drying period although moisture content decreased rapidly. Near equilibrium, however, very small decreases in moisture content cause relatively large decreases in fire endurance.

These results indicate that tests of fire endurance for gypsum plaster slabs must be conducted after the moisture content reaches equilibrium. Otherwise the test results will have little bearing on the actual fire endurance of the materials under normal use. Also, the study showed that minimum drying time for gypsum plaster slabs exposed on both surfaces to air at normal room conditions of temperature and humidity was about 15 to 20 days for each inch of thickness.



Normalized time ratios are the ratios of the equivalent fire endurance time for a one inch slab divided by the average fire endurance time for one inch specimens in equilibrium with the ambient.

[1] Fire endurance of gypsum slabs, *by J. V. Ryan. Presented during the 67th Annual Meeting of ASTM, June 1964, and to be published as part of an ASTM Special Technical Publication covering the Symposium on Fire Tests—Moisture Influence of Material Behavior.*



Standards and Calibration

Waveguide Reflectors: Frequency Range Extended

Measurement of the reflection coefficient magnitude of waveguide reflectors (mismatches) in WR137 waveguides (5.85–8.20 GHz) has been announced as a new calibration service by the Radio Standards Laboratory. A calibration service in two other waveguide sizes, WR90 and WR62, is also available.

The calibration can be performed at any frequency within the range of 5.85 to 8.20 GHz. Some degree of economy to the customer results if calibrations are requested at the selected frequencies of 6.45, 7.00, and 7.40 GHz. Measurements can be made over a range of 0.024 to 0.2, with an uncertainty of the reflection coefficient magnitude expressed as $\pm(0.0002+0.002|\Gamma|)$, where $|\Gamma|$ is the numerical value of the measured magnitude.

Microwave Noise Sources: Calibration Available in WR62 Waveguide

The Radio Standards Laboratory announces that the measurement of effective noise temperature of waveguide noise sources is now available in WR62 waveguide (frequency range 12.4 to 18.0 GHz). Suggested calibration frequencies are at 13.5, 15.0, and 17 GHz. For noise sources with an effective noise temperature of approximately 11,000 °K and a reflection coefficient magnitude of 0.09, the calibration will be reported to an uncertainty of ± 250 °K. The calibration system can measure noise sources with effective noise temperatures in the range of 1000 to 300,000 °K.

The noise source must be fitted with an output connector that is compatible with the UG 419/U waveguide flange connector. Direct current for operation of the gas discharge tube should be selected and specified within the range of 1 to 300 mA. With the noise source in an operating condition, the source must have an input reflection coefficient magnitude no greater than 0.09 (i.e., approximately VSWR$<$1.2).

Calibration of Coaxial Bolometer Units

The Radio Standards Laboratory of the NBS Institute for Basic Standards announces that services are now available for the measurement of calibration factor of nominal 50-ohm coaxial bolometer units and coaxial bolometer-coupler units. These devices have proved useful in the accurate measurement of cw rf power in coaxial systems over a range of 1 milliwatt to 10 watts. At present the service is offered for bolometer units at two frequencies only, 100 MHz and 1 GHz; for bolometer-coupler units the service is offered at 30, 100, 200, 300, 400, 500 MHz, and 1 GHz. Plans call for extension of the frequency range to at least 10 GHz, and for essentially continuous frequency coverage.

A bolometer unit includes both the bolometer element and the bolometer mount in which the element is supported. The element may be of the barretter type, consisting of a short length of silver wire of approximately 0.0001 in. diameter (Wollaston wire); or it may be the thermistor type, in the form of a bead of semiconductor material. As a metallic conductor the element has a positive temperature coefficient of resistance, as a semiconductor the coefficient is negative. The element is designed to have a resistance in the range of 50 to 200 ohms, and is made a part of a bridge circuit. The bridge provides a means of measuring the rf power absorbed by the element in terms of accurately known d-c power which is substituted for the rf power in order to restore bridge balance when the rf power is withdrawn. This d-c power is known as the substituted d-c power.

The element is supported in the bolometer mount at a position where it absorbs a maximum amount of the rf power fed into the bolometer unit. In one form a single element is used; in another, two elements are used in a symmetrical arrangement between the inner and outer coaxial conductors. It is common practice to use a Type-N connector to join the bolometer unit into the measurement system. However, several types of precision connectors are being developed by industry that will provide for greater precision in performing the calibration.

The calibration factor for bolometer units is defined as the ratio of the substituted d-c power in the bolometer unit to the rf power incident upon the bolometer unit. The calibration factor of a bolometer unit combined with a coaxial directional coupler is defined as the ratio of the substituted d-c power in the bolometer unit on the side arm of the coupler to the rf incident upon a non-reflecting load attached to the output port of the main arm.

Bolometer units are calibrated at power levels of 1 and 10 milliwatts only. Bolometer-coupler combinations are calibrated for coupling ratios in the range of 3 to 30 dB. Bolometer units should be of the fixed tuned or untuned broadband type and permanently attached to the coupler. The directional coupler should have good design features, with a directivity of at least 30 dB, and a VSWR no greater than 1.10 for the input and output ports of the main arm of the coupler.

Limits of uncertainty in determining the calibration factor of a well designed bolometer unit or bolometer-coupler unit are within 1 percent; although somewhat wider limits in the uncertainty of measurement may result for bolometer units and for bolometer-coupler units having a VSWR above 1.05.

Changes in NBS Radio Broadcasts

New frequency added by WWVH. Beginning June 1, 1965, station WWVH at Maui, Hawaii will transmit its time and frequency services at 2.5 MHz, besides the present frequencies of 5, 10, and 15 MHz. The added transmission will be modulated with the same code as other frequencies. This service is intended to improve the accuracy with which WWVH broadcasts can be received in the Hawaiian Island area.

Frequency-correction data published. Because of wide interest in accurate frequency values now available as a result of improved monitoring and control techniques, the Bureau furnishes each month frequency-correction data relating to broadcasts from radio stations WWV, WWVH, WWVL, and WWVB. The first notice exhibited data beginning January 1, 1965, and was published in the April 1965 issue of the Proceedings of the IEEE, published by the Institute of Electrical and Electronics Engineers. Subsequent data have appeared and will continue to appear each month.

Frequency. The frequencies of WWV, Greenbelt, Md., WWVH, Maui, Hawaii, and WWVL, Ft. Collins, Colo., are intended to be offset from their nominal values by -150 parts in 10^{10} during 1965. The frequency of WWVB, Ft. Collins, Colo., is not offset.

The frequencies of WWV and WWVH are kept constant at their intended values, within ± 0.5 parts in 10^{10} and ± 1 part in 10^{10}, respectively. The frequencies of WWVL and WWVB, Ft. Collins, Colo., are controlled more closely, to within ± 2 parts in 10^{11} of their intended values.

The fractional frequency deviations tabulated for WWV and WWVH are 5-day running averages of daily 24-hr values. The daily averaging period ends at 1800 UT for WWV and at 2000 UT for WWVH. The daily 24-hr values are obtained by comparisons made at the stations with transmissions from WWVL (20 kHz nominal) and WWVB (60 kHz). The fractional frequency deviations tabulated for WWVL and WWVB are 24-hr averages observed over the period ending at 1800 UT for each day listed. The measurements are made in terms of the NBS time scales, which are in turn referenced to the United States Frequency Standard (USFS) at Boulder, Colo.

Time. The time signals emitted from WWV and WWVH are in agreement with each other within 1 ms at present. Since these signals are locked to the frequency of the transmissions, a continuous departure from UT2 may occur. Differences for WWV are determined and published by the U.S. Naval Observatory. The time signals are maintained within about 100 ms of UT2 by offsetting the broadcast frequency from the USFS at the beginning of each year and by making step adjustments of 100 ms in phase on the first of a month when necessary. Because of the offset in frequency now in use, the time intervals are longer than one second (by 150 parts in 10^{10} for 1965). This system was initiated on January 1, 1960. Later, on August 10, 1963, WWVB began broadcasting time signals in accordance with this system, until January 1, 1965.

Since January 1, 1965, the time signals from WWVB give the international unit of time—the second. These signals are also locked to the broadcast frequency, which is no longer offset. A continuous departure from UT2 also occurs, and the differences are coded on the broadcasts. The time signals are also maintained within about 100 ms of UT2 by making step adjustments of 200 ms in phase on the first of a month when necessary.

Experimental studies are being made to determine the most effective technique for transmitting time from WWVL.

Frequency offsets and time adjustments from January 1, 1960, through January 1, 1963, were given in the Proceedings. On November 1, 1963, on April 1, 1964, and on September 1, 1964, there were retardations of 100 ms in time pulses to maintain close agreement with UT2. On October 1, 1964, a retardation of 1 ms was made simultaneously with an advance of 1.6 ms in Naval Observatory clocks to bring the epochs of U.S. Government broadcasts in closer agreement.

Adjustment of seconds pulses. There was no change on June 1, 1965, in phase of seconds pulses emitted from stations WWV, WWVH, and WWVB. However, the phase of time pulses broadcast by stations WWV and WWVH was retarded 100 ms at 0000 on July 1 (7:00 p.m., EST of June 30), in accordance with an announcement made by the Bureau International de l'Heure. Simultaneously the phase of time signals broadcast from station WWVB was retarded by 200 ms. No change in phase of time pulses broadcast by these stations was made on August 1.

The phase adjustments are made necessary by changes in the speed of rotation of the earth, with which the UT2 time scale is associated. The phase adjustments ensure that the pulses emitted from all stations will remain within about 100 ms of the UT2 scale. The pulses transmitted by these stations will continue to be compared with UT2 every month to determine when further adjustments are necessary.

New WWVB time code. Since July 1, 1965, Radio Station WWVB has been broadcasting time information using a level-shift carrier time code. The code is binary coded decimal (BCD) and is broadcast continuously. The new code replaces the present seconds pulses of uniform width.

The code is synchronized with the 60 kHz carrier signal. It is generated by reducing the power of the carrier by 10 dB at the beginning of each second and restoring it 0.2 seconds later for uncoded markers or binary "zeroes," 0.5 seconds later for binary "ones," and 0.8 seconds later for ten-second position identifiers and for minute markers.

Each minute the code presents time of year information in minutes, hours, and day of the year and the actual milliseconds difference between the time as broadcast and the best known estimate of UT2.

A detailed explanation of the code will be published by the Bureau at an early date.

Publications of the National Bureau of Standards

Periodicals

Technical News Bulletin, Vol. **49**, No. 7, July 1965. 15 cents. Annual subscription: $1.50; 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

CRPL Ionospheric Predictions for October 1965. Three months in advance. Number 31, issued July 1965. 25 cents. Annual subscription: $2.50; 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards.

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75. Single copy, 70 cents.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section D. Radio Science. Issued monthly. Annual subscription: Domestic, $9; foreign, $11.50. Single copy, $1.

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"Ultra Low Frequency Electromagnetic Fields" Papers

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Thermophysics Specialists To Convene in California

A conference of thermophysics specialists, under the auspices of the American Institute of Aeronautics and Astronautics, will be held September 13 through 15, 1965, at the U.S. Naval Postgraduate School at Monterey, Calif. Cosponsors of the conference are the National Bureau of Standards, the National Aeronautics and Space Administration, and the U.S. Air Force. Joseph C. Richmond of the NBS staff is on the committee making arrangements for the event, which is the sixth of its kind to be held in this country.

The purpose of the conference is to assemble engineers, physicists, and representatives of other disciplines in order that they may share ideas and experiences involving thermal radiative phenomena at temperature extremes. A better understanding of these phenomena is necessary to predict, measure, and control interior spacecraft temperatures that are governed by radiative heat transfer. Newly developed techniques and instrumentation for measuring thermal radiation will be discussed.

Over forty papers will be presented during the sessions of the conference. These sessions will deal with ablation processes and mechanisms, space environmental effects on thermal control surfaces, radiation properties, thermal modeling and interface effects, problems of thermal design revealed by satellite experiences, and lunar and near planetary thermal environments.

All persons attending the conference are required to register. Programs, registration blanks, and conference information may be obtained from the headquarters office of the American Institute of Aeronautics and Astronautics, Inc., 1290 Sixth Avenue, New York, N.Y., 10019. Preprints of the talks will be available at the time of the conference.

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